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Slow crack growth behavior of silicon nitride ceramics in cryogenic environment

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Abstract

The slow crack growth behavior of Si_3N_4 ceramics in ambient and cryogenic environment had been investigated by dynamic fatigue testing. The fracture strength was principally dependent on stress rate at 293 K while the fracture strength was comparatively independent of stress rate at 77 K. The morphologies of the fracture surface and the crack tip region were examined to correlate with the slow crack growth behavior. The experimental results revealed that the Si_3N_4 ceramics exhibits a very high resistance to slow crack growth (SCG) in cryogenic environment. The reasons for such high resistance are likely related to the local residual stress associated with either the thermal anisotropies arising from the presence of the grain boundary glass phase, or from the crack tip shielding mechanism.

Keywords: SCG; Dynamic fatigue; Cryogenic environment; Silicon nitride

1. Introduction

Silicon nitride ceramics have been intensively investigated over the past decade for their potential applications as high-temperature structural material [1–3]. Recently, scientists have found that Si_3N_4 could be a promising candidate serving in cryogenic environment [4–6]. For instance, Si_3N_4 ball bearing in liquid oxygen/hydrogen engines can operate at high speeds and stress in cryogenic temperatures and corrosive environments. Several preliminary studies have already been made on the mechanical performance of Si_3N_4 ceramics at cryogenic temperatures. Xue et al. [7] reported that the fracture toughness of Si_3N_4 ceramics increased obviously with decreasing temperature from 293 to 77 K. Wei et al. [8] found more pronounced *R*-curve behavior in Si_3N_4 ceramics at cryogenic temperatures. In fact, the use of silicon nitride ceramics as structural ceramic components is often limited by lifetimes that are controlled by a process of SCG.

SCG is the evolution of damage that temperature, stress, and environment produce in the material. It must be minimal and predictable before the structural components can be confidently applied because the accumulation of damage will ultimately dictate the performance and lifetime of the component [9–10]. However, literature studies of SCG behavior are sparse and inconclusive in the silicon nitride ceramics under cryogenic circumstance. That severely limits the use of silicon nitride ceramics in such potential environment, particularly in applications demanding long-term service. Hence, it is extremely significant to investigate the SCG behavior of the silicon nitride ceramics in cryogenic environments.

In this paper, we investigate the SCG behavior of Si_3N_4 ceramics using constant stress-rate (dynamic fatigue) testing under ambient and cryogenic environment. In order to better understand the environmentally assisted crack growth at different temperatures, the fracture surfaces and the crack propagations in the region of the crack tip were examined to correlate with the SCG behavior.

2. Experimental procedure

2.1. Sample preparation

Commercially available powders of Si_3N_4, Al_2O_3 and Y_2O_3 were well mixed in isopropanol in accordance with Si_3N_4:

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Al_2O_3:Y_2O_3 = 90:5:5 in weight by planetary ball milling. The obtained mixture was dried and sieved. Then the green body was cold isostatically pressed at a pressure of 100 MPa and sintered at 1750 °C under 0.1 MPa overpressure of nitrogen for 90 min. The densities of as sintered samples, determined by Archimedes' method, exceed 98% of theoretical density.

2.2. Dynamic fatigue test

The sintered Si_3N_4 bulk was machined to parallelepiped-shaped bars with dimension of 3 mm (thickness) × 4 mm (width) × 40 mm (length). All surfaces were ground with a diamond grinding wheel and the tensile surfaces of the specimens were carefully polished to 0.5 μm. Dynamic fatigue tests were conducted in three-point flexure with a span of 30 mm at four different loading rates (0.01, 0.05, 0.5 and 5 mm/min) using a universal testing machine (SUNS UTM4000, China) in ambient air (293 K) and liquid nitrogen (77 K). The cryogenic temperature was obtained by a test chamber filled with liquid nitrogen. The relative humidity in ambient environment was ~30%. Seven bars were tested at each loading rate and the stressing rates were then calculated from the stress–time diagram. The average data obtained were used for the subsequent SCG analysis.

The SCG behavior can be approximated by the empirical power-law relation [11]:

$$v = \frac{da}{dt} = A\left[\frac{K_I}{K_{IC}}\right]^n \tag{1}$$

where v, a, and t are crack velocity, crack length and time, respectively. A and n are the material/environment dependent slow crack growth parameters, K_I and K_{IC} are stress intensity factor and the fracture toughness of the material, respectively.

For dynamic fatigue testing, the flexural strength at a constant applied stress rates can be expressed as [12]:

$$log\sigma_f = \frac{1}{n+1} log\dot{\sigma} + \frac{1}{n+1} log[B(n+1)\sigma_i{}^{n-2}] \tag{2}$$

where σ_f is a fracture strength related to the corresponding stress rate, σ_i is an inert strength, B is a material/environment parameter. When the strength is plotted as a function of stress rate on a logarithmic scale, the SCG parameters n can be determined by a linear regression analysis based on Eq. (2).

2.3. Crack propagation characterization

Studies elucidated that the fracture surface morphologies of ceramic materials after dynamic fatigue as well as the crack tip morphologies were significant in understanding the SCG process [13]. In light of this, four specimens were fractured in four-point rectangular beam flexure with an inner span of 10 mm and outer span of 30 mm using the universal testing machine (SUNS UTM4000, China) at 293 K and 77 K. The displacement rate was 0.01 mm/min. Before testing, the samples for SEM observations were plasma etched in a gas mixture of CF_4 and O_2 (95/5 in flow rate) with a pressure of 62 Pa at a power of 220 W for 30 s. Then, each specimen was made three Vickers indentations with a load of 49 N for 15 s on the inner span of the tensile surface uniformly by a Vickers hardness tester (Tukon2500B; Wilson). The cracks induced by the indentations were carefully oriented perpendicular to the edges of a bend bar. The neighboring indentations were at least 2 mm apart to minimize interactions among adjacent cracks. After fractured from one of the indentations, two indentations were left on each remnant bar and the cracks propagated to the instability crack length [14]. The extension region in the immediate vicinity of the crack tip on the tensile surfaces and the fracture surfaces of the bend bars were observed via scanning electron microscopy (Leo 1530, Zeiss, Germany). Furthermore, the phase compositions of Si_3N_4 ceramics after the dynamic fatigue tests at different temperatures were analyzed by X-ray diffractometer (Bruker D8 Advance Diffractometer).

3. Results and discussion

Dynamic fatigue curves for Si_3N_4 ceramics at 293 K and 77 K are plotted in Fig. 1. Each data point is an average of the data of 7 samples tested at the same condition, and the standard deviation is shown by the error bar at each data point.

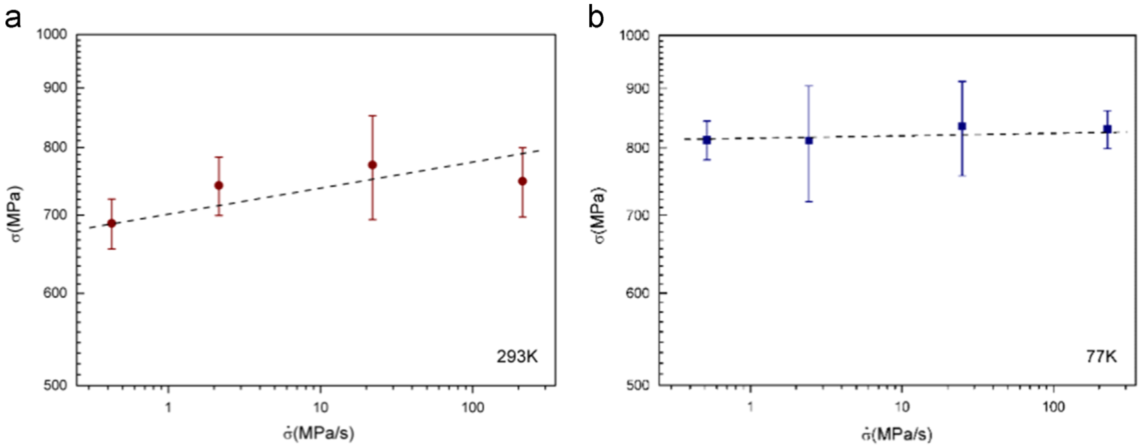

Fig. 1. Dynamic fatigue data for Si_3N_4 ceramics at (a) 293 K and (b) 77 K.